Thermal stress issues in thin film coatings of x-ray optics

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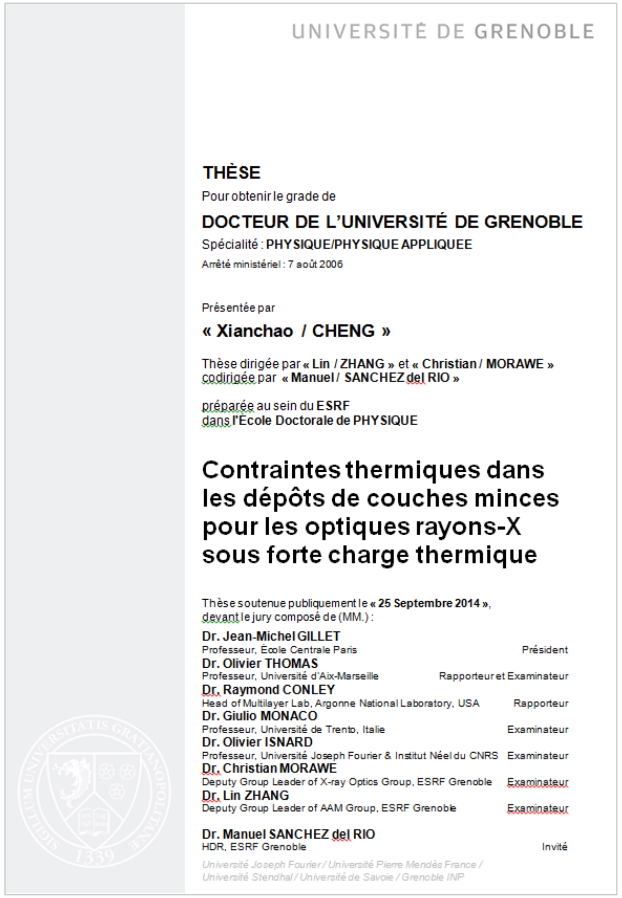
Outline

Xianchao CHENG, “*Thermal stress issues in thin film coatings of X-ray optics under high heat load*”, **PhD Thesis**, Université de Grenoble, France, 2014



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Contraintes thermiques dans les dépôts de couches minces pour les optiques rayons-X sous forte charge thermique

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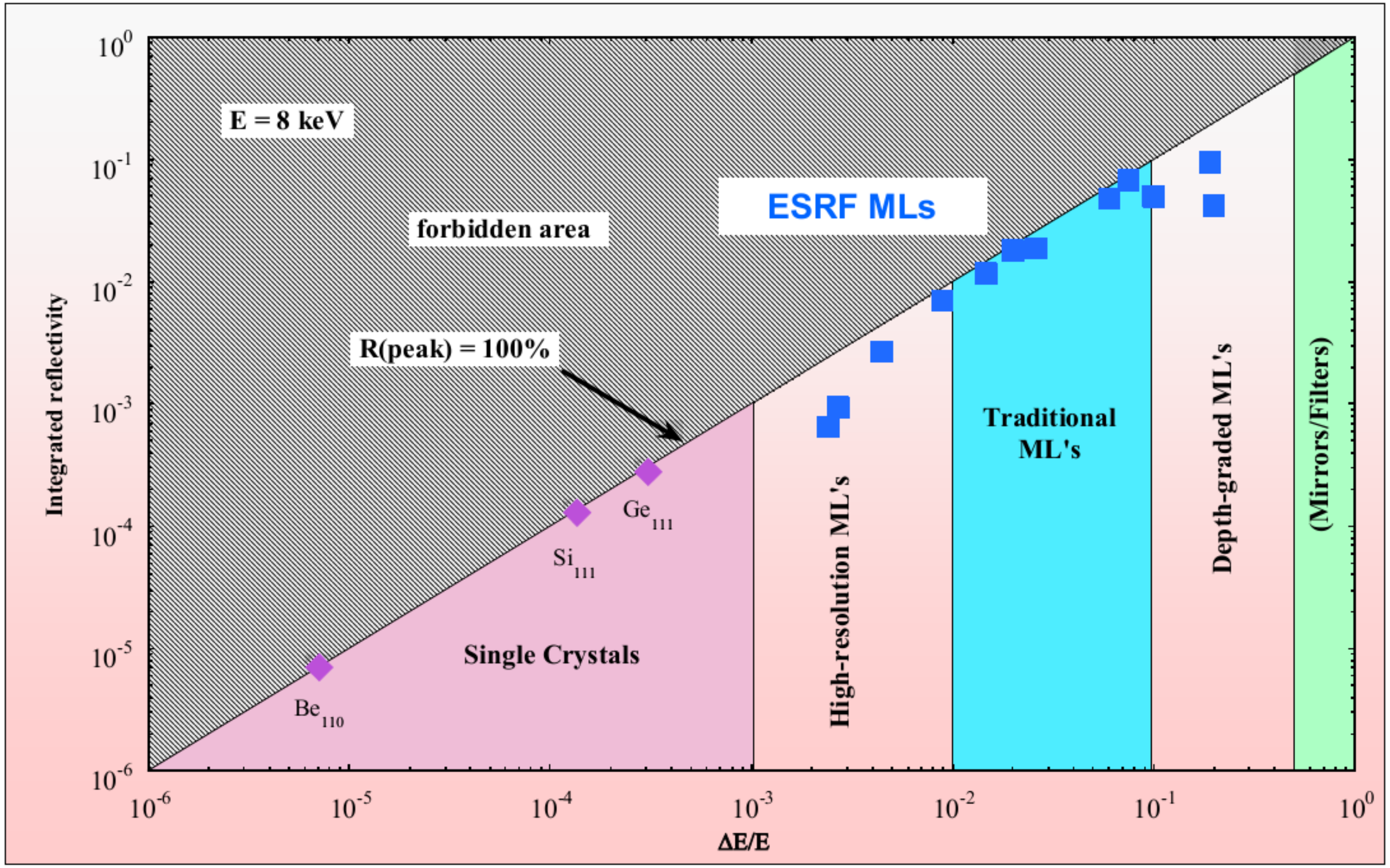
- **Introduction**
- **FE modeling of multilayer optics**
- **Thermal stress prediction in mirror and multilayer coatings**
- **Measurement: thermal deformation properties of thin coating film**
- **Summary and outlook**

Introduction – Reflective X-ray optics

Integrated reflectivity versus energy resolution



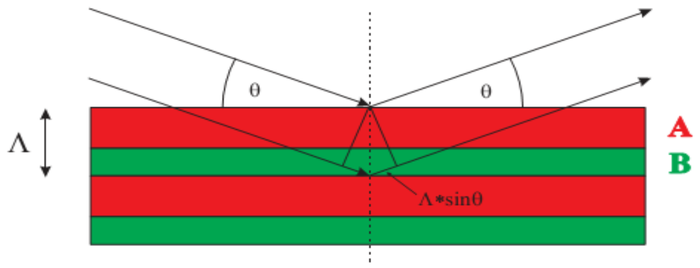
Monochromator crystal **Multilayer optics** **Mirror**

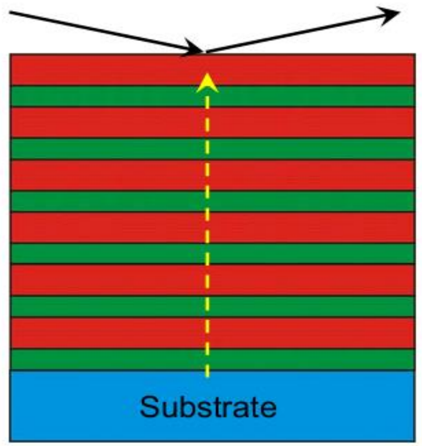
Introduction – Multilayer X-ray optics

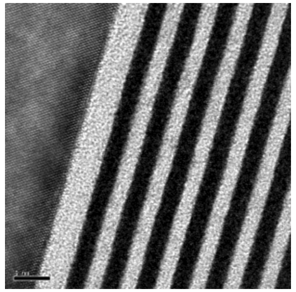
- **Multilayer structure with two types of alternative sub-layers (very thin) on a substrate. Period: Λ**
- **Multiple Bragg reflections**

$$\Lambda = \frac{\lambda}{2\sqrt{n^2 - \cos^2\theta}} \qquad \sim \text{nm}$$

- **Situated between Mirror and Monochromator for both photon flux and energy resolution**
- **Attractive alternative to crystals:**
  - High photon flux
  - Moderate energy resolution
  - Harmonics suppression
  - Reflectivity enhancement
- **Applications**
  - EUV lithography (e_{ph}=94 eV)
  - "Water window" (e_{ph}=280…550 eV)
  - Hard x-rays (e_{ph}=1…100 keV)







TEM photograph of a Ru/B_4C multilayer stack

Introduction – Heat load issues in X-ray optics

Thermal deformation

- **First mirror substrate**
  - Top side water cooling, full length illumination, optimized notches
- **Monochromator crystal**
  - Liquid nitrogen cooled Silicon crystal
  - Water cooled Diamond crystal

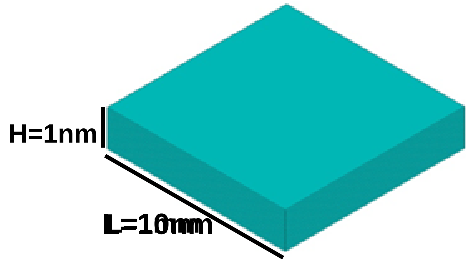
Multilayer optics (with large number of periods of thin coating) :

- **Thermal stress within the layers**
- **Thermal deformation**
  - Substrate + multiple thin layers
  - Mutual influences between the substrate and multiple thin layers ?
- **Cooling**
  - Water or Liquid nitrogen ?

→ FEA + Experiments

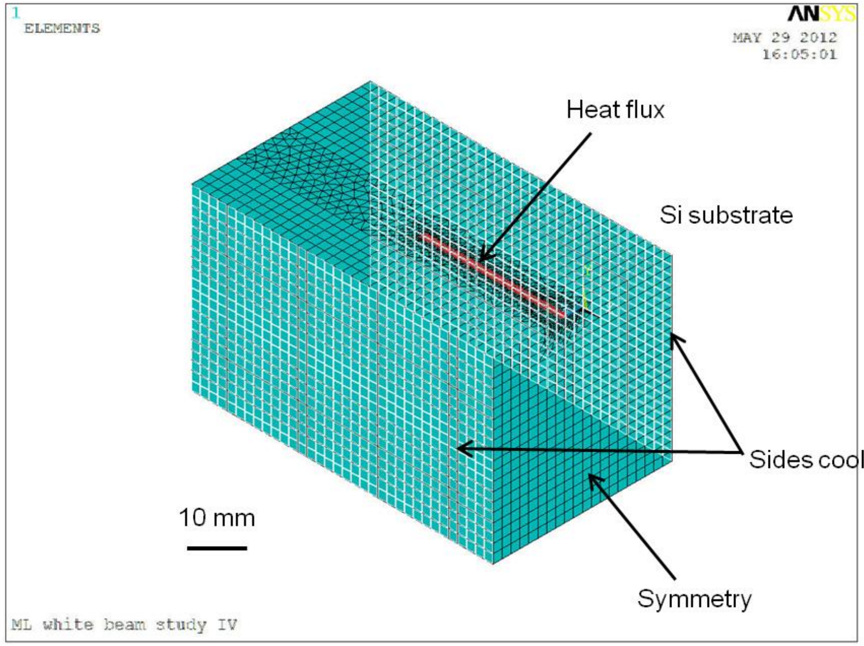
Difficulties of modelling

• High element shape aspect ratio: L/H=10^6
Program warning happened at L/H>20
➡Low solution accuracy



Element geometry, with H zoomed for clarity

• Huge number of elements
(e.g. with element size 1×10×10nm^3, more than 5×10^{15} elements just for the layer part)
➡ Impossible task for the present computers



Example FE model for substrate

The multilayer on top
•Two types of alternate materials
•Hundreds of layers
•Layer thickness several nanometers

Very high aspect ratio between the size of the optics and the layer thickness!

Finite Element Modeling of multilayer optics

- **ANSYS multilayer elements recently developed (release 12.0 and after, since 2011) for composite materials :**
  - One geometrical Shell element, multiple sub-layers (material properties, temperature or displacement)
  - Thermal analysis: **shell131** (up to 31 sub-layers)
  - Structural analysis, shell181, **solsh190**
  - The thermal and structural multilayer elements are not exactly corresponded
  - Structural model needs to be reconstructed, and temperature loading to be applied by use of array parameters
  - Various validation tests performed

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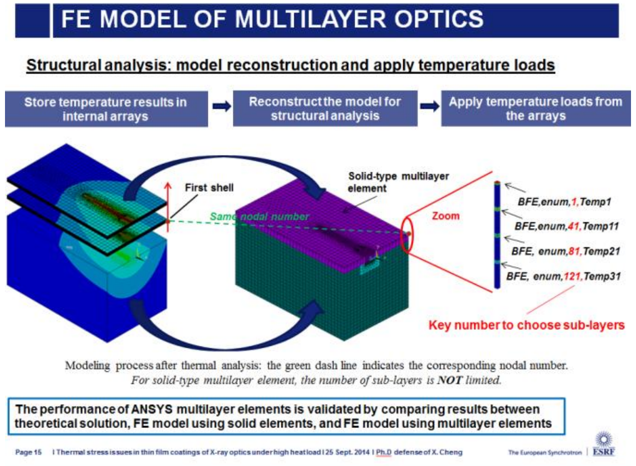


Thermal stress prediction in mirror and multilayer coatings

Mirror

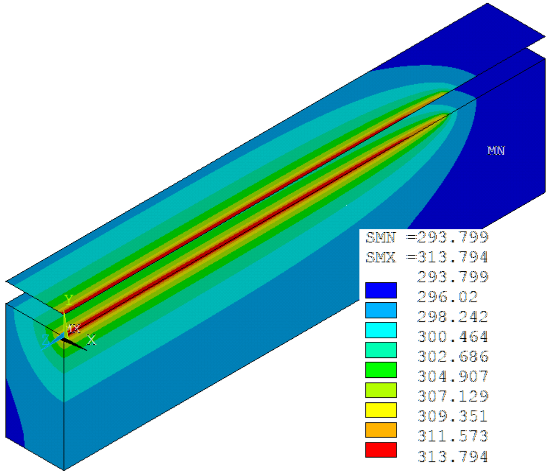
1. Water cooling
2. LN cooling

Multilayer

3. Water cooling
4. LN cooling

Thermal stress prediction in mirror and multilayer coatings

Single layer white beam mirror – water cooling



Coating: **B_4C 50 nm**

Si Substrate: 60×60×500 mm^3

Slits (H×V): 4×2 mm^2

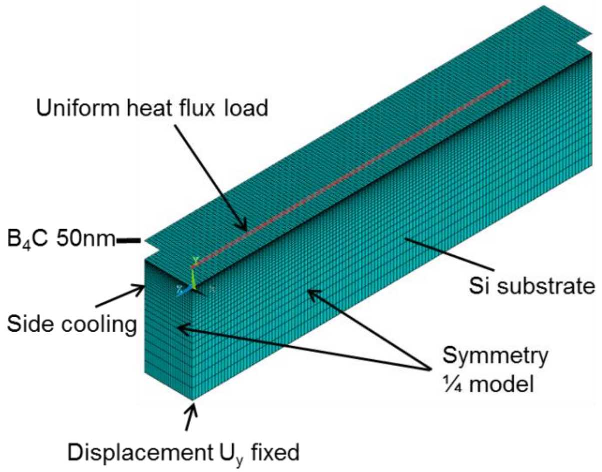
Power density: P_a=100 W/mm^2

Grazing angle: α_{inc}=5 mrad

Footprint length: 2/sin(α_{inc})=400 mm

Water cooling:

H_{cv}=0.005 W/mm^2/K, T_{cool}=293 K



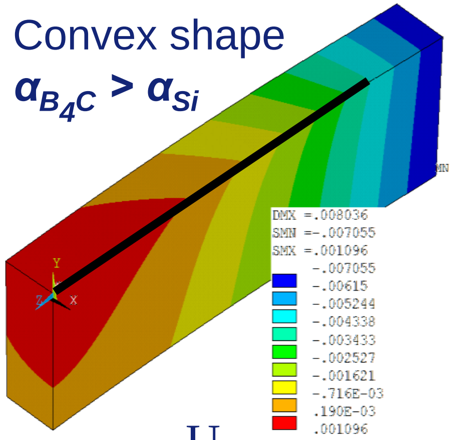
Temperature difference within the coating layer

FEA: ΔT=0.553 mK

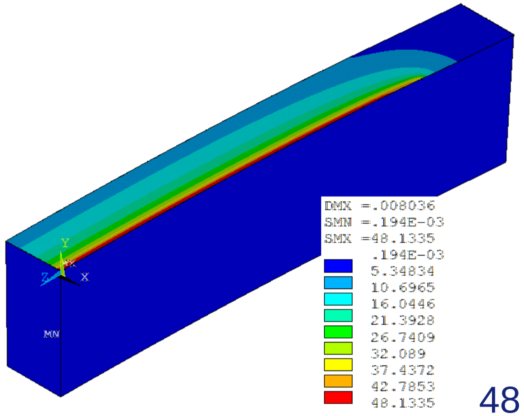
Analysis using 1D conduction: 0.595 mK

Convex shape

$\alpha_{B_4C} > \alpha_{Si}$



U_y



S_{eq}

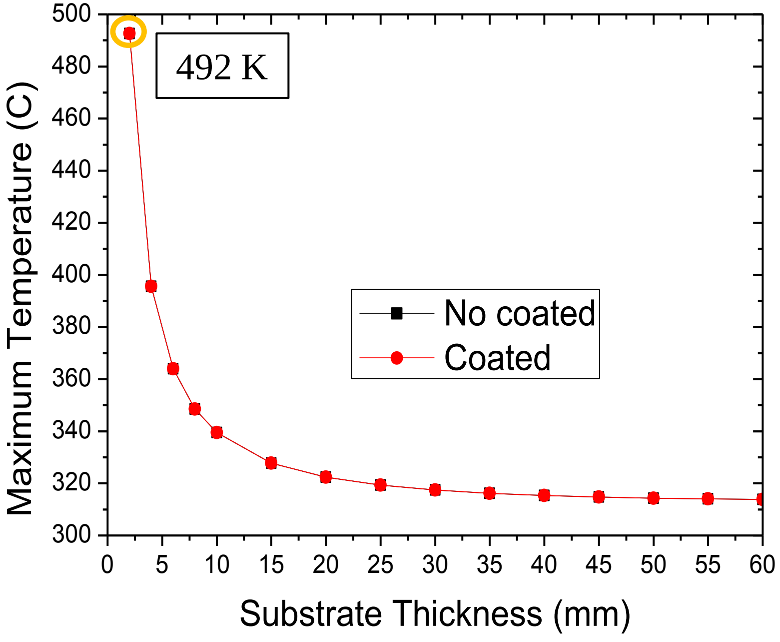
48.13 MPa

Compressive stress in the coating layer

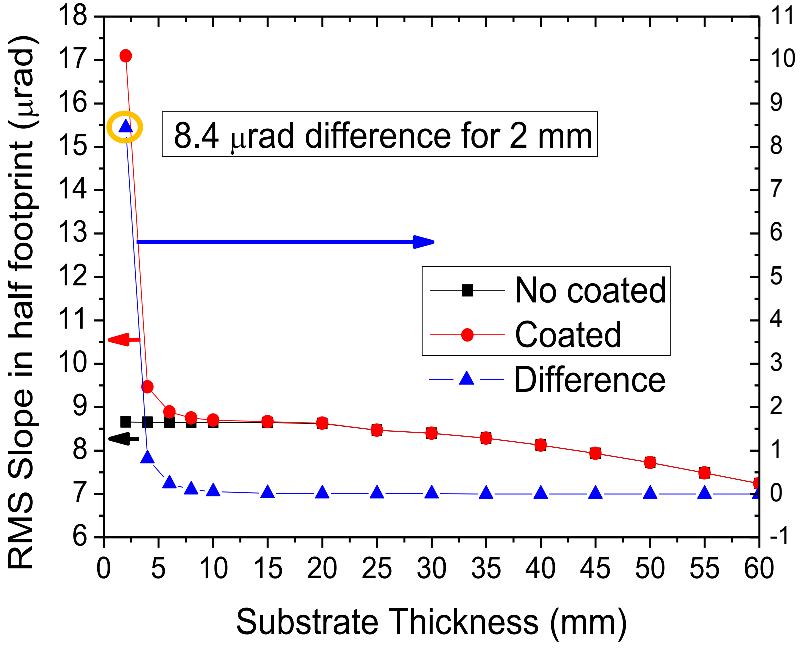
Negligible stress in the substrate

Single layer white beam mirror – water cooling

- ➢ **One interesting issue: substrate becomes thinner**



$T_{max} \sim t_{substrate}$



$Slope_{RMS} \sim t_{substrate}$

- ➢ **For $t_{substrate}$ = 2 mm, slope error change due to coating is 8.4 μrad.**

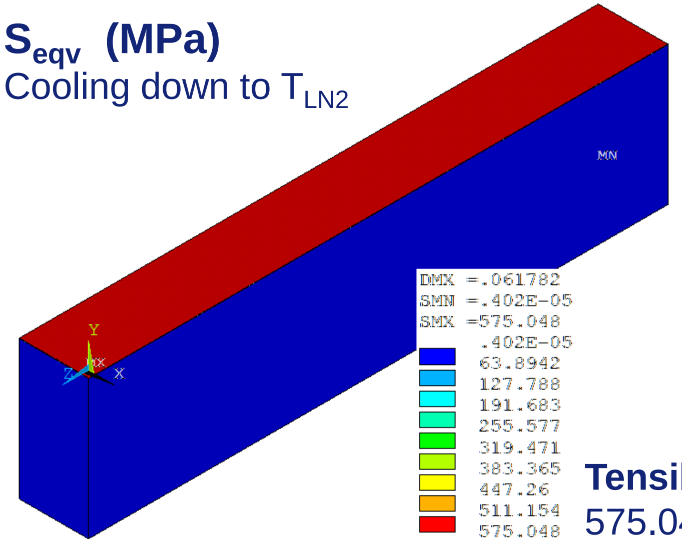
Thermal stress prediction in mirror and multilayer coatings

Single layer white beam mirror – liquid-nitrogen (LN2) cooling

h_{cv}=5000 W/m^2/K

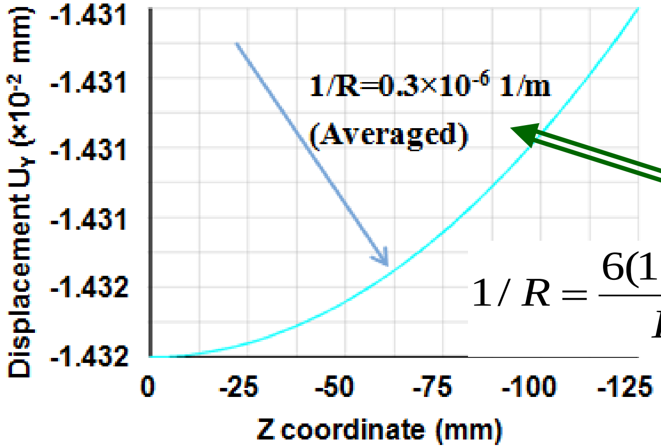
T_{cool}=80 K

S_{eqv} (MPa)
Cooling down to T_{LN2}



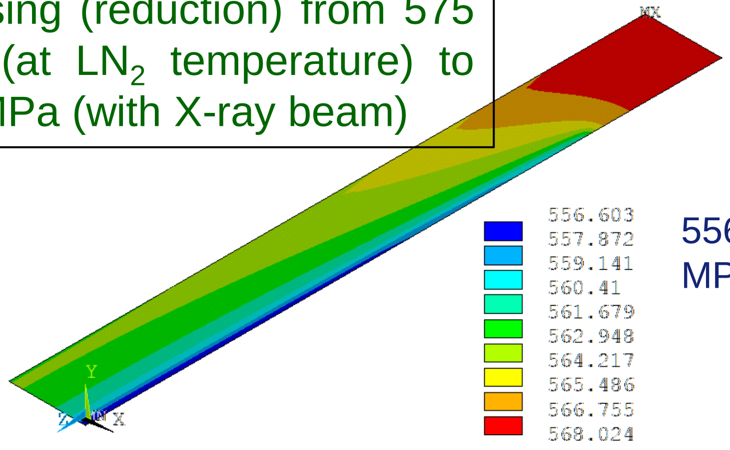
Tensile stress
575.048 MPa

$$\sigma_i \approx \frac{E_i}{1-\nu_i}(\alpha_s - \alpha_i)\Delta T = 575.1MPa$$



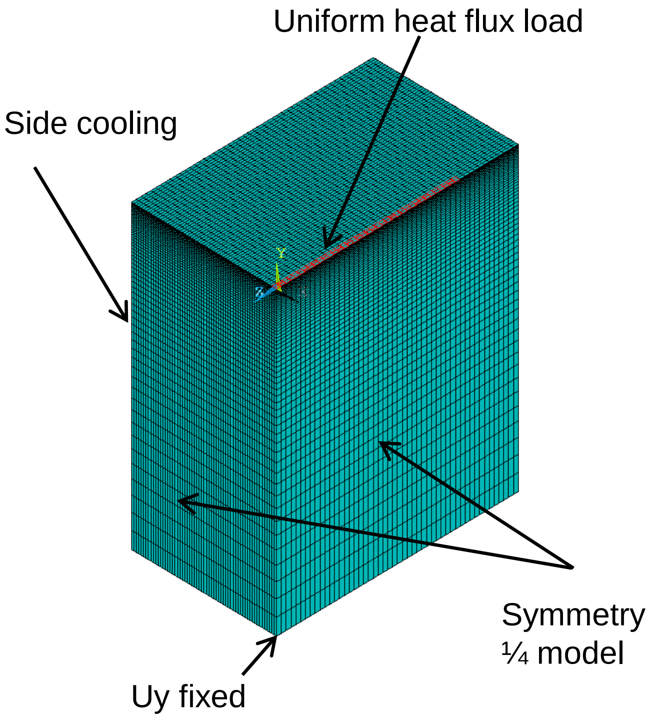
$$1/R = \frac{6(1-\nu_s)t_i}{E_s t_s^2}\sigma_i = 0.3\times10^{-6} \quad 1/m$$

Heat load leads to stress releasing (reduction) from 575 MPa (at LN_2 temperature) to 557 MPa (with X-ray beam)



556.6
MPa

Multilayer white beam monochromator – water cooling

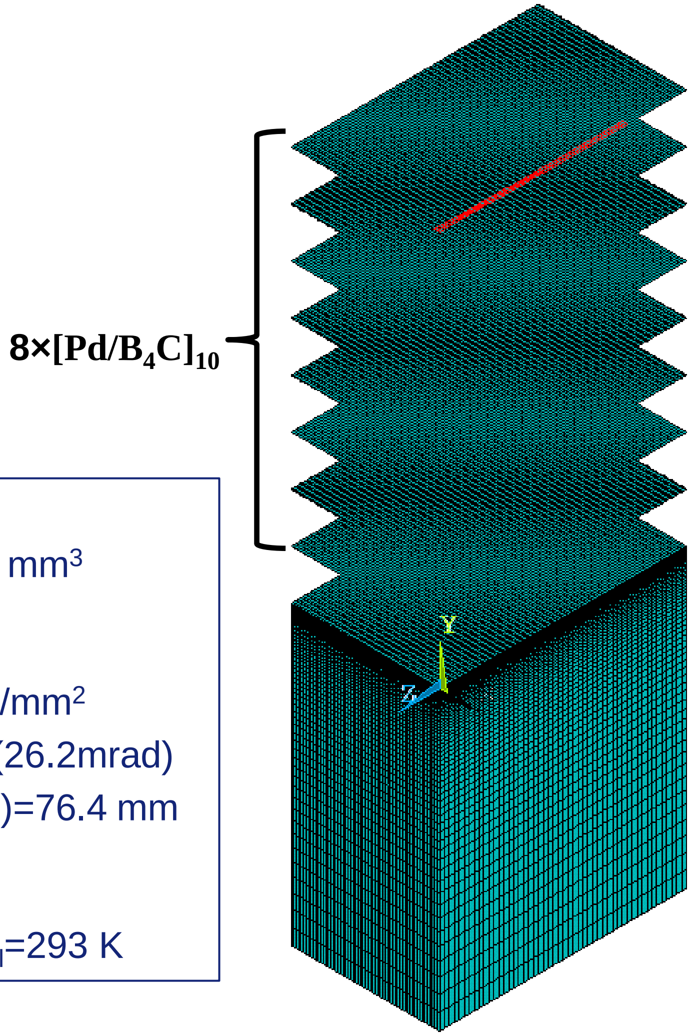


FE model (1/4) and boundary conditions

Coating: **$[Pd/B4C]_{80}$ 2 nm**
- Si Substrate: 60×60×100 mm^3

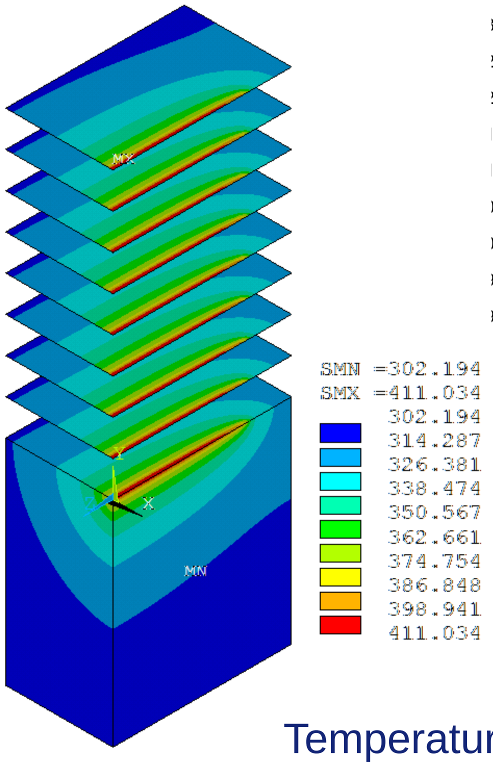
Slits (H×V): 2×2 mm^2
- Power density: P_a=200 W/mm^2
- Grazing angle: α_{inc}=1. 5° (26.2mrad)
- Footprint length: 2/sin(α_{inc})=76.4 mm

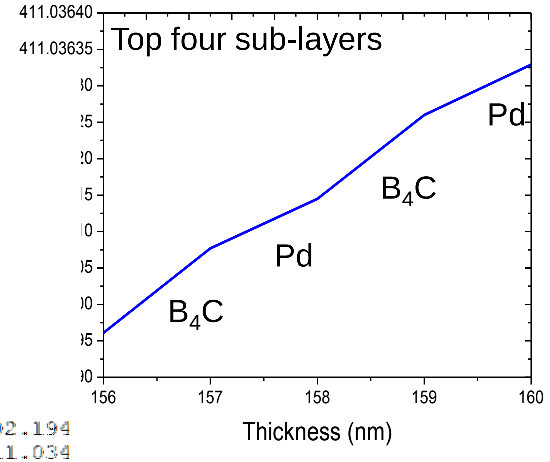
Water cooling:
- H_{cv}=0.005 W/mm^2/K, T_{cool}=293 K

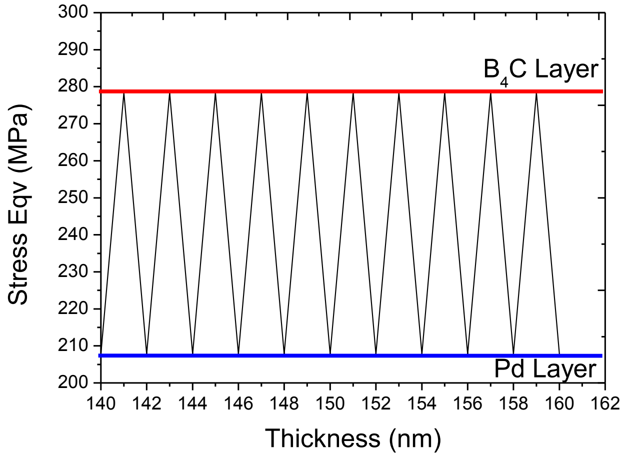


Thermal stress prediction in mirror and multilayer coatings

Multilayer white beam monochromator – water cooling



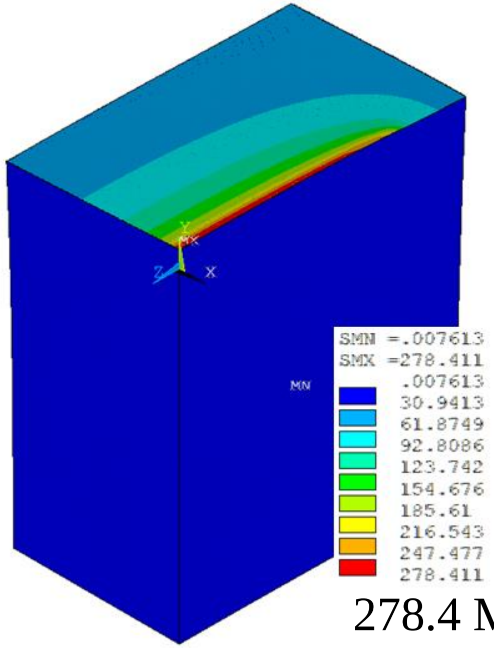




Temperature difference within the multilayer coatings

- FEA: ΔT=14.7 mK
- 1D conduction: 15.8 mK

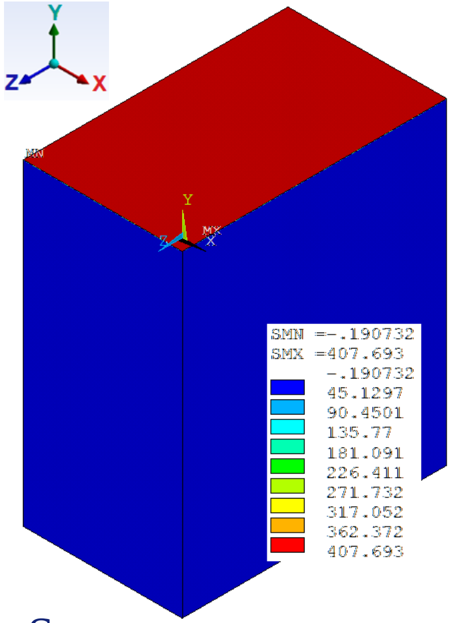
- **Compressive stress** in the coating layer
- Negligible stress in the substrate



278.4 MPa

Multilayer white beam monochromator – LN2 cooling

Uniform temperature
$T_{room} \rightarrow T_{LN2}$



S_{eq}

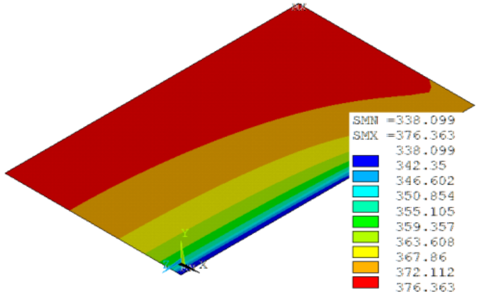
Tensile stress
408 MPa

Analytical estimation for layer stress (S_x, S_z):

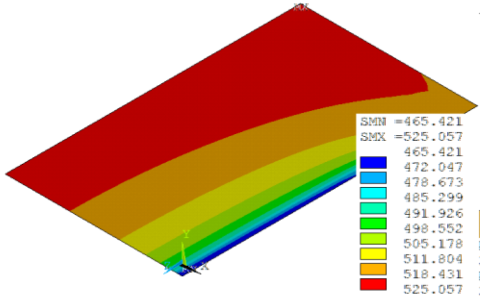
$$\sigma_i = \frac{E_i}{1-\nu_i}(\alpha_s - \alpha_i)\Delta T$$

Difference between the sub-layer and substrate i: B_4C or Pd

With X-ray beam (800 W)



S_{eq} in **Pd** sub-layer



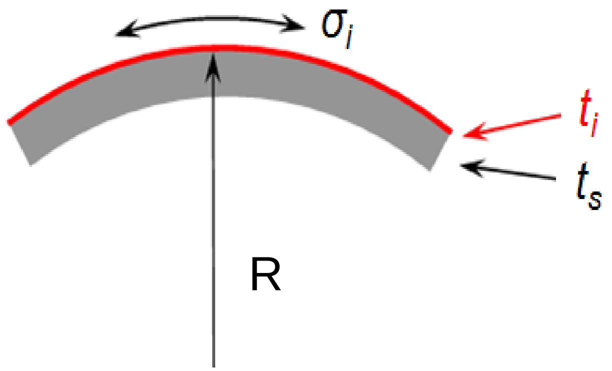
S_{eq} in **B_4C** sub-layer

Stress (MPa)	Pd sub-layer	B_4C sub-layer
analytical: $T_{room} \rightarrow T_{LN2}$	407.7	575.1
FEA: $T_{room} \rightarrow T_{LN2}$	407.7	575.0
FEA: with X-ray beam	338.1	465.4

110 MPa stress releasing with X-ray beam

Measurement: thermal deformation properties of thin coating film

- Mechanical properties of thin film are different from bulk material
- Limited data in literature for thin film
- Experimental measurements



Experimental principle

- uniform temperature change ΔT
  → curvature change Δ(1/R)
- It can be shown that

$$define \quad K = \boxed{\frac{E_i}{1-\nu_i}(\alpha_i - \alpha_s)} = \boxed{(\Delta \frac{1}{r} / \Delta T)} / [\frac{6(1-\nu_s)t_i}{E_s t_s^2}]$$

Thermal deformation properties

Measurement results

Known parameters

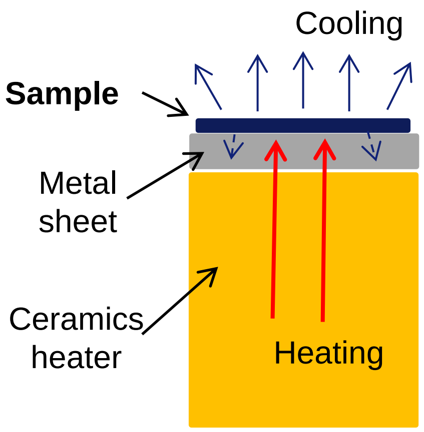
- For multilayer structure

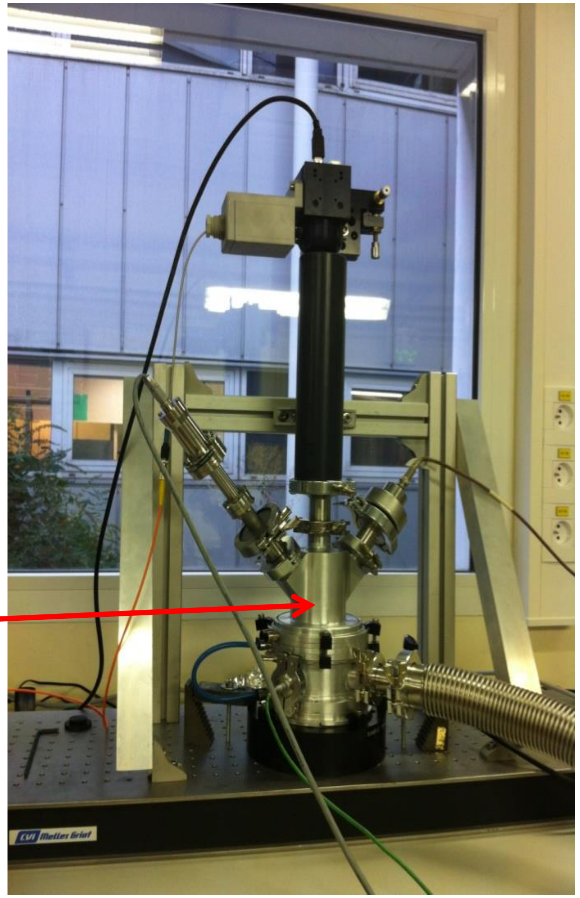
$$K = \frac{t_1 \cdot K_1 + t_2 \cdot K_2}{t_1 + t_{21}}$$

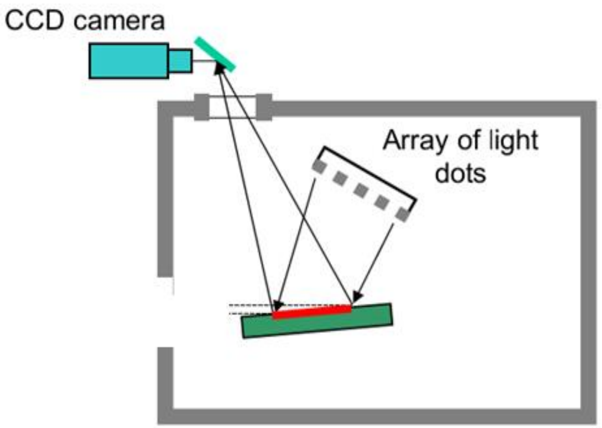
- K is a combination of parameters E, *α, ν*
- The physics meaning of the composite parameter: $\sigma_i = K \cdot \Delta T$

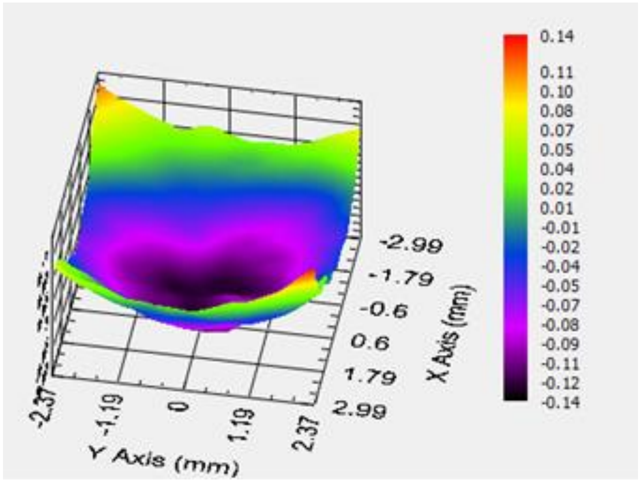
Measurement: thermal deformation properties of thin coating film

➢ Experiment setup









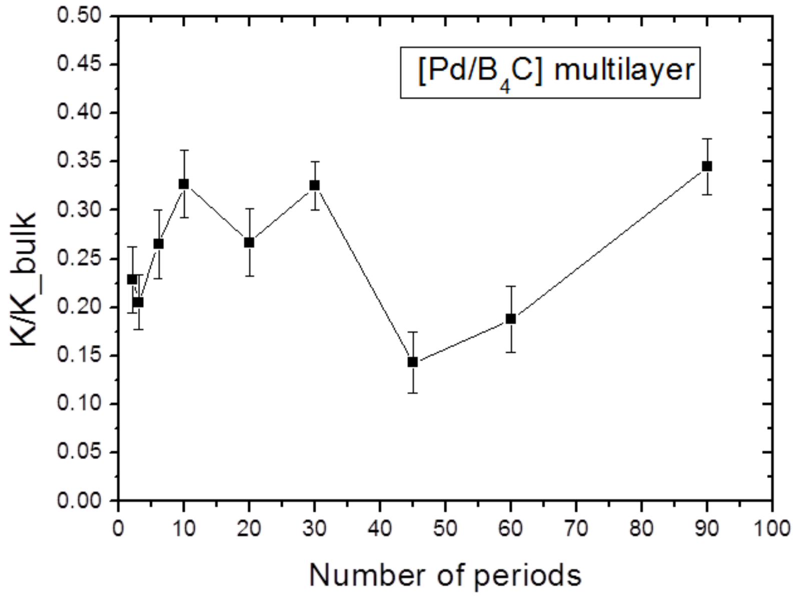
Measurement: thermal deformation properties of thin coating film

➢ Experiment results – Single layer coatings (substrate Si 200µm)

Layer	B_4C 50 nm	B_4C 20 nm	B_4C 10 nm	Pd 200 nm	Pd 100 nm	Pd 50 nm	Cr 200 nm	Cr 100 nm	Cr 50 nm
K (MPa/°C)	**0.193**	**0.874**	**0.918**	**1.322**	**1.166**	**0.749**	**0.968**	**0.509**	**0.514**
K_bulk	**1.93**			**1.63**			**1.27**		

➢ Experiment results – [Pd/B_4C] Multilayer coatings (substrate Si 200µm)

Total thicknesses of the coatings are maintained at 180nm when varying the number of periods



In all the cases, the composite parameter K of the thin coating film is **significantly smaller (1/3)** than one of the bulk material

→ Thermal stress is effectively lower

Summary

- **FEA of multilayer optics allows to study the thin coating layers**
- **Coating layers on the Mirror or multilayer optics under white beam:**
  - Negligible influence on temperature, deformation of the optics
  - Negligible influence on the stress of the substrate
- **Large thermal stress in the coating layer due to thermal mismatch between layer(s) and substrate**

$$\sigma_i = \frac{E_i}{1-\nu_i}(\alpha_s - \alpha_i)\Delta T$$

- **Water cooled Mirror or multilayer optics under X-ray beam**
  - Mostly negligible, compressive stress when using silicon as substrate (if $\boldsymbol{\alpha_i > \alpha_{Si}}$)
- **LN2 cooled Mirror or multilayer optics with Si-substrate**
  - Cooling down from T_{room} to T_{LN2} → large tensile stress (most critical case)
  - Additional X-ray beam power → additional compressive stress → globally stress released in the coating layers
- **Thin-coating material properties:**

$$K = \frac{E_i}{1-\nu_i}(\alpha_i - \alpha_s)$$

  - Smaller composite parameter K than bulk material
  - Smaller stress in coating layers than expected with bulk material properties
- **Intrinsic stress from deposition (0.5~1.0 GPa compressive)**
  - LN2 cooling induced tensile stress should offset this Intrinsic stress from deposition